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REPORT OF INVESTIGATIONS/1991

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Optical Rock Dust Meter Field Evaluation

By M. J. Sapko, N. Greninger, and H. Perlee

UNITED STATES DEPARTMENT OF THE INTERIOR



BUREAU OF MINES



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Manuel Lujan, Jr., Secretary

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	μm	micrometer
ft	foot	Ω	ohm
g	gram	pct	percent
g/cm ³	gram per cubic centimeter	st	short ton
h	hour	st/d	short ton per day
in	inch	V	volt
kΩ	kilo ohm	wt pct	weight percent

OPTICAL ROCK DUST METER FIELD EVALUATION

By M. J. Sapko,¹ N. Greninger,² and H. Perlee¹

ABSTRACT

The U.S. Bureau of Mines has developed a portable meter for measuring the component concentrations in binary dust mixtures whose constituents have different optical reflectivities. The meter has been successfully tested with coal dust-rock dust mixtures taken from coal mines in Europe and the United States and fluorinated-carbon mixtures that change from carbon black to pure white during fluorination. Additional study has also substantiated the theoretical expressions used in the design of the meter.

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INTRODUCTION

The U.S. Bureau of Mines has developed a prototype handheld optical reflectivity meter that provides a rapid method for determining the composition of binary powders whose components have different optical reflectivities, such as black coal and light-colored rock dust. Although originally intended for use in measuring in situ rock dust content of coal dust-rock dust mixtures in coal mines, the meter can also be used with other dust mixtures.

The meter's operation is based on the measurement of the intensity of near-infrared light reflected from the

surface of a dust sample consisting of a mixture of highly reflective and poorly reflective particles. The amount of radiation reflected from the sample surface increases with the increase in concentration of the highly reflective component in the mixture. Unfortunately, the accuracy of the meter depends not only on the differences in the optical reflectivities of the binary components but also on the particle-size distribution and the moisture content. However, this is not a problem since the meter can be calibrated without detailed knowledge of the particle-size distribution.

ACKNOWLEDGMENTS

The authors wish to thank Geneva College, Beaver Falls, PA, Electrical Engineering Department for their many technical discussions regarding the research; E. Bazala, who conducted the numerous experiments and R. Helinski, who constructed the electronic equipment.

Special thanks goes to G. Klinefelter from USX's Cumberland Mine, Cumberland, MD, for helping in the evaluation of the rock dust meter and Robert Cortese for his technical comments and suggestions regarding this report.

BACKGROUND

To prevent coal dust explosions in underground coal mines, rock dusting is required by law in the United States. Although rock dust (limestone) is the most commonly used inertant, other light-colored dusts, such as gypsum and dolomite, are used. The law requires that without methane that there be at least 80 wt pct total incombustible content (TIC) in the dust deposited in returns and 65 pct elsewhere in the mine, except the first 40 ft from the face where there is no requirement if the last open crosscut is not within 40 ft of the face. TIC includes all inertants, e.g., water, ash, rock dust, etc., whereas the rock dust meter measures just the rock dust content. If the last open crosscut is within 40 ft from the face, then the inert content of the dust up to the last open crosscut must also meet the appropriate 65 or 80 pct requirement.

Inspectors for the Mine Safety and Health Administration (MSHA), in determining compliance with the law, currently collect samples of deposited dust from areas in question. In making a judgement as to whether samples are to be collected, visual inspection is often used—poor rock-dusted areas along the entry appear dark, and well rock-dusted areas appear light. When samples are collected in any given mine, they are usually collected every 200 ft of entry every 2 months. The preponderance of samples that are collected are relatively dry. The inspector screens the samples through a 10-mesh sieve and sends about 200 g of the sieved sample to MSHA's Mount Hope

facility in West Virginia for chemical analysis. Typically, it requires about 2 weeks between taking the sample and receiving the results of the analysis. Meanwhile, the mine operators are forced to rely on visual inspection of the rock-dusted areas from which the samples have been taken in determining whether to commence immediately with the rock dusting. To alleviate this problem, the Bureau developed a permissible portable rock dust meter (fig. 1), which permits an in situ determination of the rock dust content of coal dust-rock dust mixtures.

Results of previous bench tests, using laboratory prepared rock dust samples, showed that the meter was accurate to within ± 1 wt pct rock dust over the range of 0 to 100 wt pct rock dust.³

Since these early bench studies were conducted, additional work has been completed using coals from West German and British mines and studies conducted using marble, dolomite, and gypsum as the inertant. In addition, a prototype meter was also loaned to USX's Cumberland Mine for field evaluation and one also to Allied Chemical Corp., Metropolis, IL, for their evaluation as a means to monitor their carbon fluoridation process. A complete description of the results of those studies is reported in the following sections.

³Pinkerton, J., H. Perlee, and M. Sapko. Monochromatic Reflectance Meter. Part II Conference Record of 1986 IEEE Ind. Appl. Soc. Annu. Meet., 1986, pp. 1582-1589.

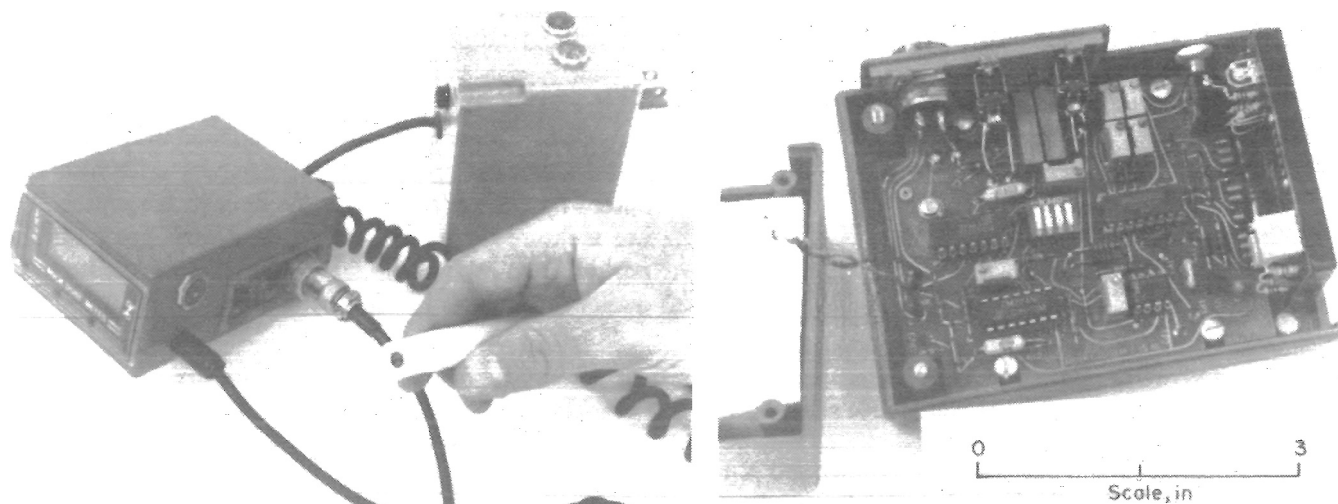


Figure 1.—Rock dust meter.

METER THEORY AND EXPERIMENTAL PROCEDURES

METER THEORY

Details regarding the design, operation, and bench testing of the reflective meter (fig. 1) can be found in the reference in footnote 3. The meter operates on the theory that homogeneous mixtures of highly reflective rock dust and much less reflective coal dust exhibit near-infrared surface reflectivities in accordance with the expression,

$$\frac{r_s - r_c}{r_i - r_c} = \frac{f}{f + K(1-f)}, \quad (1)$$

where

$$K = \frac{\rho_i d_i}{\rho_c d_c} \quad (2)$$

and r is the dust layer surface reflectivity, f is the mass fraction of rock dust, ρ is the particle density, d is the surface-weighted-mean particle diameter⁴, and the subscripts c , i , and s refer to coal, inert, and sample dusts, respectively. Although K , through equation 2 is a function of the particle density and the mean particle diameter, the expression is only approximately true since it is not clear from the theory what type of mean diameter (number weighted mean, surface weighted mean, mass weighted mean, etc.) is appropriate. Furthermore, even if the mean were known for the particle size, the particle-size distributions are normally not available, so the value of K

must be determined through calibration procedures. The quantity on the left side of equation 1 is given the symbol ϕ and will in the sequel be referred to as the normalized reflectivity. The meter determines ϕ by measuring the surface reflectivities of the various dusts by means of a probe that houses a light emitting diode (LED) monochromatic light source and a PIN diode receiver. Since the surface reflectivities are proportional to the intensity of the light reflected from the dust surface, which is in turn directly proportioned to the PIN current, i , ϕ is actually calculated by the relation in equation,

$$\phi = \frac{i_s - i_c}{i_i - i_c} \quad (3)$$

Previous bench tests have demonstrated that the meter has a measurement accuracy of better than ± 1 wt pct rock dust,⁵ over the range 0 to 100 wt pct rock dust when tested with laboratory prepared rock dust-coal dust samples.

FIELD EVALUATION PROCEDURES

Mathies Coal Mine

The Mathies Coal Mine, Washington, PA, covers approximately 33 square miles, and in good times its coal output averages 11,000 st/d per shift. The coal contains about 8 wt pct ash.

⁴Woods, T., M. Sapko, and H. Perlee. Analog Optical Rock Dust Meter. Paper in Proceedings, Mining Industry Committee of IEEE Industry Society 23rd Annual Meeting. Pittsburgh, PA, 1988.

⁵Work cited in footnote 3.

Twelve 200-g grab samples, were collected from the Mathies Coal Mine, divided into two groups and each of the six samples were in turn divided into two parts. One half of each grab sample was analyzed by the Bureau and the other half at MSHA's Mount Hope facility. Samples of the mine's pure coal dust and pure rock dust were also collected at the mine by Bureau personnel for subsequent analysis. In addition, rock dust content measurements were conducted at the mine site. In all cases, the samples were dried before making a measurement. The sample's TIC was measured at the Mount Hope Laboratory using gravimetric and low temperature ashing analysis (LTA) heating to 125° C for 24 h with a reported accuracy of ± 5 wt pct rock dust and alcohol volumetric analysis with an accuracy of ± 5 wt pct.

Analysis of the samples at the Bureau involved X-ray analysis to confirm that the inert dust was limestone and total moisture analysis using (1) the Computrac⁶ Corp. analyzer, (2) conventional oven drying at 105° C for 24 h using duplicate samples, and (3) LTA using duplicate samples. Moisture in the samples is comprised of bound moisture that is internal to the coal particles and surface moisture that fills the interstructural region. The former moisture has little effect on the dust optical reflectivity, but the latter has a significant effect and must be removed before testing.

Cumberland Coal Mine

USX's Cumberland Coal Mine covers approximately 10,000 square miles, and in good times its coal output

averages 12,000 st/d per shift. The coal contains 9.6 wt pct ash and 4.0 pct moisture.

Ninety grab samples of dust were collected from all parts of the mine and sealed in plastic bags by USX employees. Each grab sample contained about 100 g of dust. Chemical analysis of the samples were also conducted by USX employees at the Cumberland Mine. TIC was measured using the alcohol volumetric procedure. A portion from each of the samples was also processed with one of the Bureau's rock dust meters.

Allied Chemical Facility

The Allied Chemical Corp., which operates a carbon fluorination facility to convert black carbon into a white fluorinated product, asked the Bureau about the feasibility of using the Bureau meter to monitor the quality of their product during various stages of the reaction. Twelve samples of Allied's dust, which included pure carbon black, partially fluorinated carbon dust, and the final fluorinated product, were forwarded to the Bureau along with analysis results accurate to within ± 0.1 wt pct F. The pure carbon black sample and the white final product sample, containing 64 wt pct F, were used to calibrate the Bureau's optical reflectivity meter. Following the Bureau's initial feasibility study, the Bureau loaned Allied an instrument for further evaluation of the meter as a means for monitoring their fluorination process.

RESULTS

MATHIES COAL MINE

The Bureau's rock dust meter measures the rock dust content in mine dust samples. The mass fraction of rock dust (f) along with the ash (A) content in weight percent and the moisture content (M) in weight percent for the pure coal was used to compute the corresponding TIC value in weight percent for the sample based on equation 4 so as to compare with the Mount Hope Laboratory result,

$$\text{TIC} = 100 f + (1 - f) (A + M). \quad (4)$$

The total moisture content of the six samples, as measured by the Bureau, ranged from 0.4 to 14.2 wt pct using the Computrac analyzer and from 0.4 to 13.7 using conventional oven drying. Figure 2 summarizes the results of the reflectivity tests. The results show that rock dust

meter 1 consistently reads higher than rock dust meter 2. The data also shows unusually large scatter compared with previous measurements. It's not clear whether the meter was faulty or the sample contained impurities such as clay.

CUMBERLAND COAL MINE

The rock dust content of the Cumberland Mine samples, which were measured by using both the Bureau's rock dust meter and USX's volumeter, are compared in figure 3. It shows good agreement—less than ± 2 wt pct error at a 0.05-confidence level.^{7,8} USX estimates that the use of the Bureau's rock dust meter reduced their cost for sampling and analyzing of their mine dust samples by 65 pct. Furthermore, USX found the Bureau's rock dust meter to be simple and quick to use.

⁷The statistical nomenclature and calculations used in this report can be found in footnote 8.

⁸Natrella, M. G. Experiment Statistics. U.S. Natl. Bur. Stand. Handb. 91, 1963, 257 pp.

⁶Reference to specific products does not imply endorsement by the U.S. Bureau of Mines.

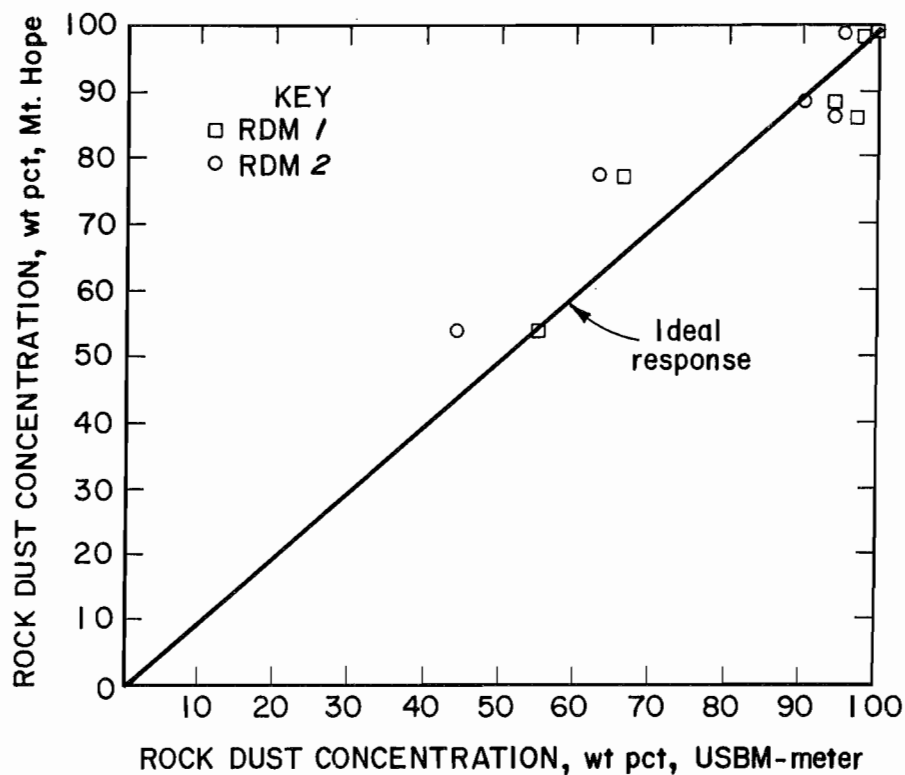


Figure 2.—Comparison of Mount Hope Laboratory LTA and analysis and Bureau rock dust meter using two different meters and six dust samples taken from Mathies Coal Mine.

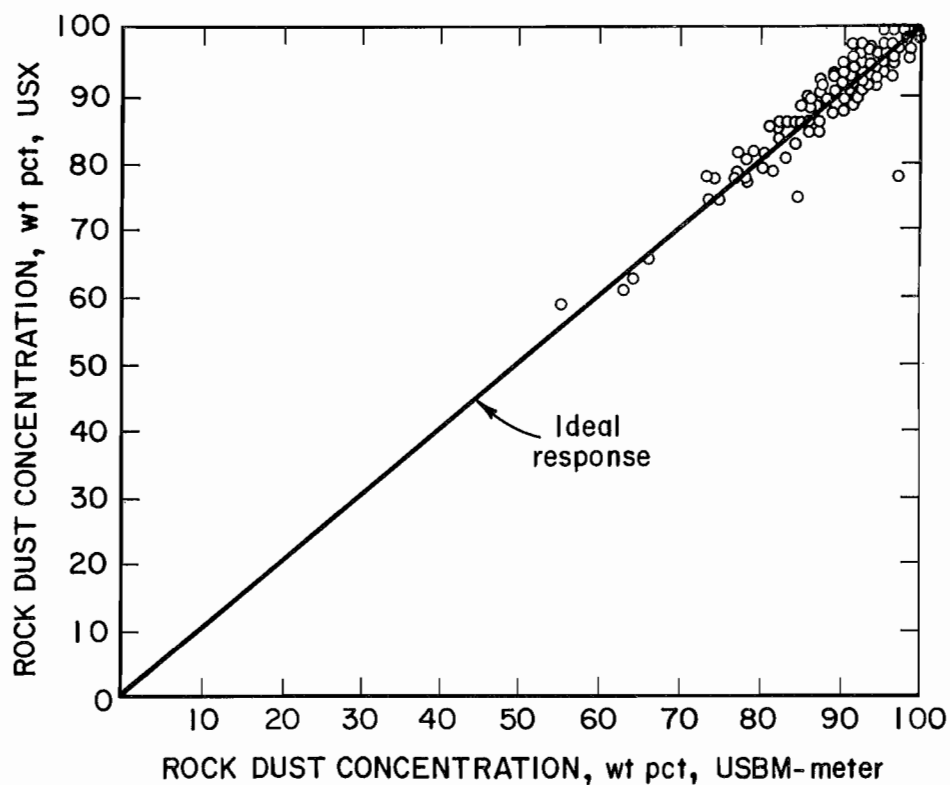


Figure 3.—Comparison of USX volumeter and Bureau rock dust mixtures taken from USX mines.

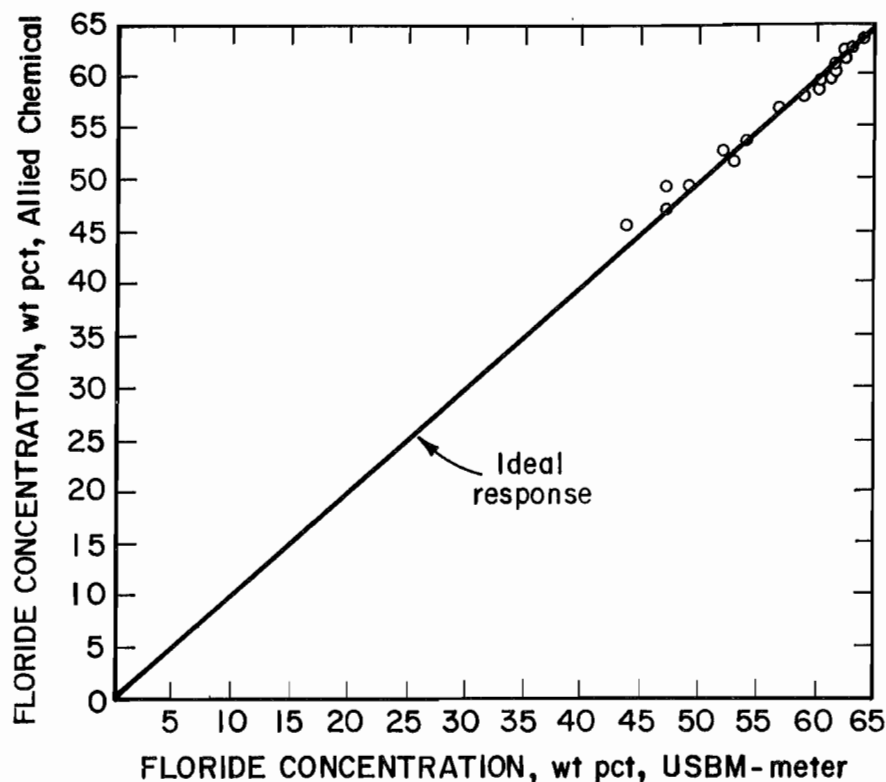


Figure 4.—Comparison of Allied Chemicals analysis and Bureau rock dust meter values for fluorinated product.

ALLIED CHEMICAL FACILITY

Figure 4 shows a plot of the fluorocarbon content measured by Allied's standard analytical procedure and

measured by the Bureau's reflectivity meter. This data show that the meter results and the conventional analysis agreed to within ± 1 wt pct at the same confidence level.

DISCUSSION

K-VALUE

It is important to examine the K-values obtained for dust mixture prepared with coal samples taken from various domestic and foreign coal mines and other combustion inertant dusts to ensure that the meter has the range to function properly in all these mines. With the exception of the British coal dust-rock dust samples, which were acquired from the Rawdon and Cadley mines in the United Kingdom as grab samples, all the other mixtures were synthetically prepared in the Bureau's laboratory from bulk coal, taken from the mine, crushed, sieved, and mixed with rock dust. Before mixing the sieved coal dust with rock dust, the size distribution of the dust was obtained using a sonic sieve and a Coulter counter. Similar procedures were followed in preparing other inert dust mixtures.

VARIANCE WITH COAL SOURCE AND SIZE

Figure 5 shows a schematic of the circuit used to measure the relative surface reflectivity of dust mixtures using a United Detector Technology Inc. PIN 3CDP sensor. (See work cited in footnote 3 for extensive details concerning its construction and operation.) Figure 6 shows plots of the measured normalized reflectivities of the British dust mixture as a function of the mass fraction of rock dust. It also shows the best fit to equation 5, viz,

$$\phi = \frac{f}{f + K(1 - f)} \quad (5)$$

As can be seen from figure 6 and the values of the standard deviation about the regression, s_ϕ , listed in

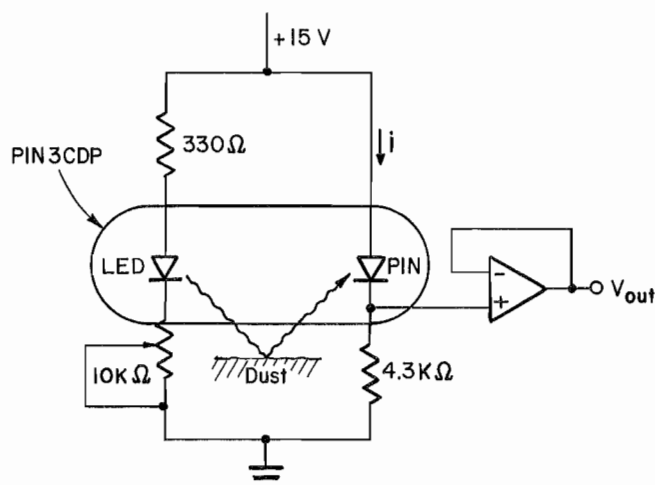


Figure 5.—Circuit for measuring relative surface reflectivity of dust mixtures.

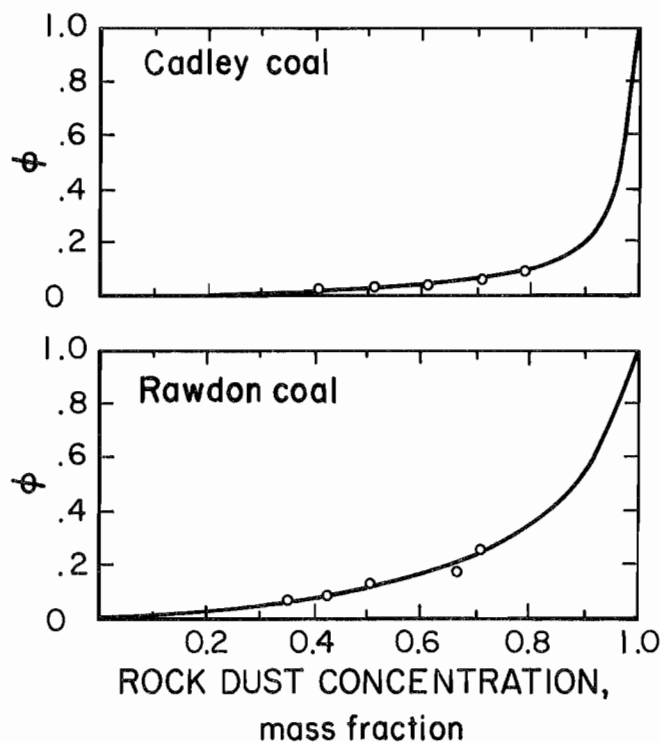


Figure 6.—Normalized reflectivity as function of mass fraction rock dust for two British coal mines.

table 1, equation 5 fits the Rawdon, B, and Cadley, C, mine samples closely. Table 1 shows a comparison of regressed \tilde{K} -values (designated in the table as \tilde{K} to distinguish it from the theoretical value of K as defined in equation 2) obtained for dust samples prepared from West German (A) and British (B and C) coal mines and

prepared from bulk coal taken from U.S. coal seams (F, G, D, and E) that was crushed, sieved, and mixed with 12 μm mean-diameter limestone dust. A discussion of these findings and other entries in table 1 is presented in the section on inerting dusts.

Table 1.—Measured \tilde{K} -values obtained for various coal dust and limestone dust mixtures from various mines

Coal source	N	d_c , μm	d_i , μm	\tilde{K}	S_ϕ	\tilde{K}/K
A ¹ ...	3	14	15	32.0 ± 2.0	0.005	(²)
B	1	NA	NA	$7.8 \pm .9$.02	(²)
C	1	NA	NA	38.0 ± 2.0	.003	(²)
D	3	26	12	$8.0 \pm .2$.01	8.0 ± 0.3
E	3	16	12	$8.0 \pm .6$.01	$5.0 \pm .2$
F	3	16	12	$13.0 \pm .5$.01	$8.0 \pm .2$
G	3	22	12	$8.9 \pm .8$.01	$4.7 \pm .1$

d_c Surface-weighted mean diameter of combustible particle.

d_i Surface-weighted mean diameter of inert particle.

N Number of measurement replications, including preparation of mixtures.

NA Not available.

S_ϕ Regression.

¹Prepared using stone dust provided by source mine.

²These values are not shown because \tilde{K} -values could not be obtained.

NOTE.—Confidence intervals correspond to a confidence level of 0.05.

INERTING DUSTS

Table 1 shows that the European dust samples yield significantly higher values of \tilde{K} than those recorded for U.S. mines. The authors attribute this to the higher concentration of fines in the European coal dust. Table 1 shows the mean particle diameters for the U. S. and European mines are statistically the same, but the European coal dust exhibits a broader size distribution and therefore more fine dust than U. S. coal dust.

In addition to limestone, three additional inerting materials were examined in these studies, namely marble, gypsum, and dolomite. Although the latter two materials are referred to as gypsum and dolomite, they are actually a mixture of gypsum and dolomite with limestone (table 2). Each of the three dusts were separately mixed with Pittsburgh pulverized coal (PPC) dust having a 22- μm mean diameter, and the resulting mixtures were measured with the rock dust meter as previously described. Table 2 shows the results of these measurements. According to the theory, i. e., equation 2, the \tilde{K} values for these mixtures should be proportional to the product of the inert dust solid density, and the particle's mean diameter or conversely, \tilde{K}/K should be constant.

Table 2.—Measured K-values for various inerts mixed with 22- μ m mean diameter G coal dust

Inert dust	ρ_i , ¹ g/cm ³	d_i , μ m	\tilde{K}	$K/[\rho_i d_i]$	\tilde{K}/K
Dolomite ² . . .	2.84	21	17.0 \pm 2.0	0.28 \pm 0.04	8.0 \pm 1.0
Gypsum ³ . . .	2.31-2.33	12	9.0 \pm .7	.27 \pm .04	9.2 \pm .7
Limestone . .	2.68-2.76	12	8.9 \pm .8	.16 \pm .01	4.7 \pm .1
Marble	2.60-2.84	17	13.0 \pm 1.0	.28 \pm .04	8.1 \pm .6

¹Weast, R. C. Handbook of Chemistry and Physics. CRC Press, 51st ed., 1971, p. F1.

²70 wt pct CaCO₃, 30 wt pct dolomite.

³40 wt pct CaCO₃, 60 wt pct gypsum.

Indeed, with the exception of G coal, tables 1 and 2 show that the latter is true, and figure 7 shows that within statistical error, \tilde{K} is a linear function of d_i . Shown in figure 8 and table 3 are the measured K-values for three G coal and rock dust mixtures prepared from 12- μ m mean diameter limestone dust and G coal dust of 5-, 17-, and 22- μ m mean diameter. Since the limestone dust diameter and solid density of the G coal dust (1.3 g/cm³) is fixed in these mixtures, theory, i.e., equation 2, dictates that the product of \tilde{K} and the coal particle diameter should be constant, or equivalently, the data in a \tilde{K} versus d_c plot should lie on a hyperbola. Indeed, the results listed in table 3 and figure 8 suggest that the product of \tilde{K} and d_c is constant, supporting the theory.

Table 3.—Measured K-values for various sized G coal dust mixed with 12- μ m mean diameter limestone dust

d_i , μ m	\tilde{K}	$\tilde{K}d$	\tilde{K}/K
5	33.4 \pm 0.9	165 \pm 5	6 \pm 1.0
17	10.0 \pm .4	150 \pm 6	5 \pm .5
22	8.9 \pm .8	195 \pm 20	8 \pm .8

Figure 8 also includes the K-values for the coal dust-limestone dust mixtures listed in table 2. The A coal limestone value (not shown in the figure) does not fit the theory, but this is to be expected since the mixtures were prepared using the source-mine's limestone dust whose particle-size distribution in this instance is not known.

Tables 1, 2, and 3 show that the ratio of the estimated K-values, i.e., \tilde{K} , to the theoretical K-value, i.e., \tilde{K}/K , ranged from 1 to 14. Apparently, the theoretical expression, i.e., equation 2, for K is incorrect. Most likely, the assumption in the derivation of equation 2, which incorporates the effect of particle size on the light

scattering through a single parameter, viz d , is an over simplification. On the other hand, one can't argue with the fact that equation 2 fits the measurements closely, viz better to within ± 1 pct (see work cited in footnote 4), as long as the parameters are determined via the method of least squares. The theory needs to be re-examined using more realistic transport concepts.

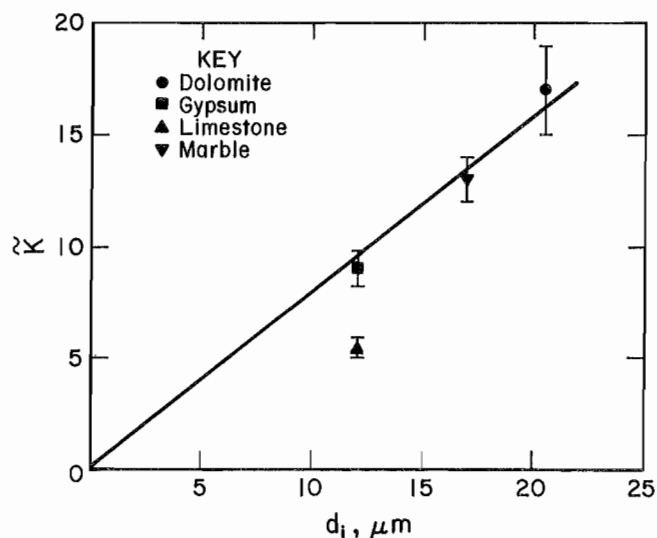


Figure 7.—K-values for various G coal dust-inert dust mixtures as function of inert dust surface-weighted mean diameter using 22 μ m PPC dust.

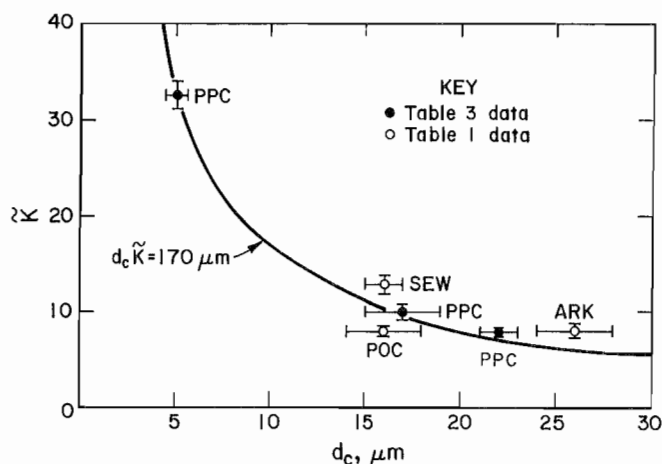


Figure 8.—K-values for various coal dust-limestone dust mixtures as function of coals surface-weighted mean diameter prepared with 12 μ m limestone dust.

CONCLUSIONS

The Bureau has developed a permissible rock dust meter for the rapid in situ determination of rock dust content, which addresses a longstanding need for a simple and rapid way of characterizing the flammability hazard of mine dust deposited on the floor, ribs, and roof in returns, along conveyor belt roadways, and other passageways in underground coal mines.

The device is simple, portable, and requires only a small representative sample of dry mine dust. Testing results, which were based on optical reflectivity measurements, correlated well with those based on LTA from MSHA's Mount Hope Laboratory. The instrument can be used to monitor the rock dust content in separate floor and rib dust samples.

The adoption of the meter for use underground will be of benefit to both government inspection agencies and the mining industry. In the majority of instances, it should enable the inspector to render a judgement on whether a region has been adequately rock dusted. If a region needs additional rock dusting, the use of the rock dust meter in situ in most instances will quickly identify the need. The judicious use of the meter by both government and industry can significantly strengthen safety and increase the efficiency of rock dusting.

A prototype version of the Bureau's meter was approved by Pennsylvania's Department of Environmental Resources for use at USX's Cumberland Mine. The use of the meter at this mine has resulted in not only a savings in analysis costs but also in time. MSHA is proceeding with the integration of the meter in the mining environment. The device is being commercialized by Ocenco Inc., Blairsville, PA, in the United States.

Separate from the mining industry, the optical reflectivity meter has already found application in the analysis of chemical dusts. A prototype version of this meter has already saved the Allied Chemical Co. considerable money in maintaining quality control in the manufacture of fluorocarbon dusts.

Results of the experiments appear to confirm that the K parameter for the optical reflectivity of binary dust mixtures is proportional to the inert dust concentration and inversely proportional to the coal dust supporting the theoretical expression for K . However, for reasons unknown, the theoretical value for K appears to be lower than the measured value by an order of magnitude.

APPENDIX.—ABBREVIATIONS AND SYMBOLS USED IN THIS REPORT

A	ash content in pure coal, pct	S	standard deviation
d	surface-weighted-mean particle diameter, μm	S_ϕ	regression
f	rock dust inert mass fraction, 1	TIC	total incombustible content, pct
i	electrical current, A	ϕ	normalized reflectivity
K	dimensionless parameter, 1	ρ	particle density
LTA	low temperature ashing		<i>Subscript</i>
\tilde{K}	regressed value K		
M	inherent moisture content in pure coal, pct	c	combustible
N	number of replications of the measurement	i	inert
r	dust layer surface reflectivity	s	sample